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(54) An ethylene polymer useful as a lubricating oil viscosity modifier.

(57) Ethylene polymer useful as viscosity modifier comprises ethylene, alpha-olefin, nonconjugated diene and, optionally, cationically polymerizable mono-olefin.

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1 AN ETHYLENE POLYMER USEFUL AS A LUBRICATING OIL VISCOSITY
2 MODIFIER

3

4

5   It is well known that refined petroleum oils gen-
6 erally exhibit substantial changes in viscosity with tem-
7 perature. The viscosity index ("V.I.") is a measure of the
8 slope of the temperature-viscosity curve. It is preferred
9 that a lubricating oil, e.g., automobile lubricating oil,
10 exhibit a "flat" V.I. curve. The desired V.I. characteristic
11 is generally achieved by adding oil soluble polymers to oil.
12 For many years the preferred polymer additive was polyiso-
13 butylene.
14   Recently, specialty ethylene-propylene copolymers
15 have been developed and are now widely used as V.I. improv-
16 ers. Since lubricating oils are used in a wide range of
17 applications, the market requires a variety of grades of such
18 polymers having differing degrees of "thickening effect" so
19 as to permit the formulation of lubricating oils having
20 different viscosities and "shear stability indicies". Such polymer
21 grades may be prepared by direct synthesis, the molecular
22 weight grades being determined by the polymerization pro-
23 cess, or the different molecular weight grades can be pro-
24 duced by degradation of an ethylene-propylene copolymer so
25 as to produce lower molecular weight fractions.
26   The patent literature is replete with many publi-
27 cations dealing with ethylene ter-and tetrapolymers con-
28 taining one or more types of dienes introduced for a variety
29 of reasons including a means for introducing unsaturation,
30 thereby providing a means for crosslinking the polymer.
31   In the case of viscosity index improvers, cross-
32 linking is neither a necessary nor desirable characteristic
33 of the polymer. Illustrative of patents dealing with un-
34 saturated ethylene ter-and tetrapolymers is U.S. Patent No.
35 3,790,480. Polymers of ethylene, C_3-C_{18} higher alpha olefins
36 and two classes of dienes are taught, the dienes having
37 double bonds of the same or different polymerizability. In

1 one class of dienes represented by 1,4-hexadiene, only one
2 of the double bonds is readily polymerizable by the catalyst
3 used. In another class of which 2,5-norbornadiene is repre-
4 sentative, both double bonds are polymerizable utilizing the
5 polymerization process of the patent. It is taught that the
6 preferred viscosity indexes improvers are ethylene tetra-.
7 polymers wherein both classes of double bonds are used.

8   Presumably, superior properties are achieved be-
9 cause use of a diene with two active double bonds results in
10 long chain branching with a concomitant increase in bulk
11 viscosity of the polymer without any significant increase in
12 intrinsic viscosity or thickening efficiency. Increased
13 bulk viscosity facilitates the manufacture and storage of the
14 polymer. The catalyst used for polymerization is a Ziegler
15 type catalyst. Both double bonds of the 2,5-norbornadiene
16 are polymerizable by the Ziegler catalyst. The other diene,
17 1-4 hexadiene, however, has only one Ziegler catalyst poly-
18 merizable double bond. Hence, the polymers include a
19 minor amount of unsaturation.

20   Unsaturation in a polymeric viscosity index im-
21 proving oil additive is generally undesirable since the
22 unsaturated moiety introduces a site through which chemical
23 reactions can occur under the conditions of use of the
24 lubricating oil. Such reactions are undesirable since they
25 cause changes in the viscosity of the lubricating oil. On
26 the other hand, branched saturated ethylene tri or tetra
27 polymers have desirable properties as viscosity modifiers.
28 :

29   It has surprisingly been found that substantially
30 saturated, long chain, branched ter-and tetrapolymers of
31 ethylene can be prepared using a non-conjugated diene polymer
32 by selecting as the polymerization initiator a catalyst
33 system which is both a coordination catalyst and a cationic
34 polymerization catalyst. The preferred coordination cata-
35 lysts are Ziegler catalysts known to be useful in the
36 preparation of ethylene-propylene-non-conjugated diene
37 terpolymers. The cationic polymerization catalysts are

1   either conventional cationic polymerization catalysts or are
2   catalyst species which, in conjunction with the coordination
3   catalyst, initiate cationic polymerization.
4   The preferred monomers are ethylene propylene and
5   5-ethylidene-2-norbornene. The preferred catalyst system is
6   VCl_4 or $VOCl_3$, in combination with $Al_2Cl_3Et_3$.
7
8   This invention relates to a polymer comprising the
9   reaction product of ethylene, an alpha-olefin and a non-con-
10 jugated diene which has utility as a viscosity modifier.
11 More particularly it relates to saturated ter-and tetra
12 polymers of ethylene, an alpha olefin and at least one non-
13 conjugated diene wherein the diene has a first double bond
14 polymerizable in the presence of a coordination catalyst and
15 a second double bond which is cationically polymerizable.
16   Not wishing to be bound by theory, it is believed
17 that a polymer of the ethylene, alpha-olefin and non-con-
18 jugated diene is formed wherein the coordination catalyst
19 polymerizable diene is incorporated into the backbone with
20 subsequent coupling of these chains involving the cationi-
21 cally polymerizable double bond. This coupling produces a
22 long chain branch in the polymer molecule. Of course, it is
23 probable that some degree of simultaneous reaction of both
24 double bonds occurs. However, since only small quantities
25 of non-conjugated diene is used, as compared to ethylene and
26 other alpha olefins, the mass effect mitigates in favor of
27 its incorporation into the backbone first. In any event, the
28 resultant polymer is a predominately saturated, long chain,
29 branched oil soluble polymer of high bulk viscosity and low
30 intrinsic viscosity.
31   The alpha-olefins suitable for use in the practice
32 of this invention are linear and branched C_3-C_{18} alpha-
33 olefins. The preferred alpha-olefins are C_3-C_8 linear alpha-
34 olefins. The most preferred alpha-olefin is propylene.
35   Illustrative non-limiting examples of such alpha-
36 olefins are propylene, butene, pentene, hexene, heptene,
37 octene, nonene, decene, dodecene, 2-methyl butene-3, 2-

1 methyl pentene-4, 2-methyl hexene-5, 2-ethyl hexene-5 etc.

2 The dienes suitable for use in the practice of this

3 invention are non-conjugated dienes having one double bond

4 which is coordination catalyst polymerizable and one double

5 bond which is cationically polymerizable. Illustrative non-

6 limiting examples of such non-conjugated dienes are 2-methyl

7 hexadiene-1,5; 2-methyl heptadiene-1,6; 5-methylene-2-nor-

8 bornene, 5-ethylidene-2-norbornene, 2-methyl norbornadiene,

9 5-isopropenyl-2-norbornene, 5-methallyl -2-norbornene,

10 5(2'-methyl-1-propene)-2-norbornene, 5-methyl vinyl-2-

11 norbornene,3-methallyl cyclopentene, and 3(2'-methyl-1-

12 propenyl) cyclopentene and dicyclopentadiene.

13 A fourth monomer which is a cationically poly-

14 merizable monoolefin such as isobutene may be included in the

15 polymerization medium. All of the monomers must be hydro-

16 carbons.

17 The polymerization is advantageously carried out

18 in solution. Suitable solvents for the polymerization re-

19 action are hydrocarbon or chlorinated hydrocarbon solvents

20 which are solvents for both the polymer and monomer. Illus-

21 trative examples of such solvents are hexane, methyl cyclo-

22 hexane, cyclohexane, pentane, isopentane, heptane, tetra-

23 chloroethylene, toluene, benzene, and so forth.

24 The catalyst system of this invention comprise (1)

25 a coordination catalyst and (2) a catalyst which is a ca-

26 tionic polymerization initiator or (3) a compound which in

27 conjunction with (1) or (2) generates a cationic catalyst.

28 Illustrative examples of the coordination catalyst of this

29 invention are those catalysts known generally as Ziegler

30 catalysts. These Ziegler catalyst comprise, for example,

31 VCl_4, $VOCl_3$, or $VO(OR)_3$ wherein R is a hydrocarbon of 1 to

32 8 carbon atoms, e.g., trialkoxyvanadate , in conjunction

33 with a cocatalyst wherein the cocatalyst is an aluminum alkyl

34 i.e., AlR_3 wherein R is as previously defined, or an alkyl

35 aluminum halide in which the number of alkyl groups is equal

36 to or greater than the number of halogens, i.e., $R_mAl_nX_p$

37 wherein X is halogen, R is as previously defined, n is an

integer, m+p=3n and m>p, e.g., $Et_3Al_2Cl_3$ or Et_2AlCl.

The term "cationic initiator" as used in the specification and claims means a catalyst which at least to some degree, initiates cationic polymerization. It may be necessary to improve the catalyst efficiency by using a cationic promoter with the cationic initiator. Suitable cationic initiators are HCl, $AlCl_3$ or an alkyl aluminum halide in which the number of halogens is greater than the number of alkyl groups, i.e., $R_rAl_sX_t$, wherein R and X are as previously defined and r, s, and t are integers of positive values and r=3s-t, for example, $EtAlCl_2$. The cationic initiator is optionally utilized in conjunction with a cationic promoter which is for example a tertiary alkyl halide, a benzyl chloride or a benzyl bromide. Illustrative examples of the tertiary alkyl halide are tertiary butyl chloride, 2-ethyl-2-chloro propane, 2-methyl-2-chlorohexane; and so forth.

As described herein the catalyst system of this invention comprises a coordination catalyst in conjunction with a catalyst which initiates cationic polymerization which comprises either a cationic initiator or a cationic initiator plus a promoter. While it will be readily recognized that the cationic promoters of this invention, alone, are never cationic polymerization catalysts by themselves when used in conjunction with particular cocatalysts of the Ziegler catalyst they can initiate cationic polymerization.

As used in the specification and claims, the term "cationic polymerization catalyst" means (1) a catalyst which of itself initiates cationic polymerization, e.g., cationic initiator (2) a cationic initiator which in conjunction with a cationic promoter exhibits improved catalytic activity, and initiates cationic polymerization or (3.) a cationic promoter which in conjunction with the cocatalyst of a Ziegler catalyst initiates cationic polymerization.

Where the cationic initiator is $AlCl_3$ or $RAlX_2$ where X is chlorine or bromine, a cationic promoter is not

1   required, but may be used to improve cationic activity.
2   Where the cocatalyst of the Ziegler catalyst is an alkyl
3   aluminum halide, as defined, e.g. $Et_3Al_2Cl_3$, the cationic
4   promoter alone in conjunction with the appropriate Ziegler
5   catalyst is a suitable catalyst system to initiate both
6   polymerization reactions.
7   In the Ziegler catalyst the ratio of co-catalyst to
8   catalyst is defined in terms of the mole ratio Al/M wherein
9   M is V or Ti. Al/M is preferably 2 to 25, more preferably
10 3 to   15, most preferably   4 to   7, e.g., 5.
11   The molar ratio of cationic initiator to Ziegler
12 catalyst is preferably .1 to 20, more preferably .5 to
13 15, most preferably 1 to 10, particularly preferably 2 to 8, eg. 3.
14
15   The amount of cationic promoter is based on the
16 amount of cocatalyst or cationic initiator used and hence,
17 is defined by the ratio P/Al wherein P represents the
18 cationic promoter P/Al can be for example .1 to 10, pre-
19 ferably 0.3 to 5, more preferably .5 to 2, eg., 1.
20
21   Table I presents non-limiting, illustrative exam-
22 ples of the catalyst system of this invention.

TABLE I

Ziegler Catalyst	Catalyst Co-Catalyst	Al/M	Cationic Initiator	Initiator* I/M	Catalyst Promoter	P/Al
VCl_4	$Et_3Al_2Cl_3$	5	HCl	3	-	-
"	$AlEt_3$	5	HCl	4	-	-
"	$Et_3Al_2Cl_3$	5	-	-	t-butyl chloride	5
$VOCl_3$	Et_2AlCl	5	HCl	4		
"	$Al_2Et_3Cl_3$	5	HCl	-	-	-
"	$Al_2Et_3Cl_3$	8	$EtAlCl_2$	-	benzyl chloride	2
"	Et_2AlCl		HCl	6	-	-
"	Et_2AlCl		$EtAlCl_2$	-	t-butyl chloride	2
"	$Al_2Et_3Cl_3$	5	$EtAlCl_2$	3	t-butyl chloride	1
"	$Al_2Cl_3Et_3$	5	$EtAlCl_2$	-	-	-

* Ratio of cationic initiator to M

As used in the specification and claims the term "Thickening Efficiency" (T.E.) means the ratio of the weight percent of a polyisobutylene having a Staudiger molecular weight of 20,000, required to thicken a solvent extracted neutral mineral lubricating oil, having a viscosity of 150 SUS at 37.8°C, a viscosity index of 105 and an ASTM pour point of 0°F, to a viscosity of 12.4 centistokes at 98.9°C, to the weight percent of a test copolymer required to thicken the same oil to 12.4 centistokes at 98.9°C.

Mooney Viscosity measures were performed in accordance with ASTM D-1646 (ML 1+8 (100°C)).

The term "shear stability index" (SSI) as used in the specification and claims means the percent reduction of the polymer viscosity after it is subjected to sonic breakdown. The viscosity of the polymer is determined before and after exposure to sonic breakdown and the SSI is recorded as the percent reduction in viscosity.

The advantages of the instant invention may be more readily appreciated by reference to the following examples.

EXAMPLE I

A solution polymerization is carried out in a continuous flow stirred reactor in the manner shown in Table II, Run A. The polymer formed had a sufficiently low molecular weight, and thus thickening efficiency, so that it had a shear stability index ("SSI") of 18% as compared to 30% for conventional ethylene propylene copolymers having a thickening efficiency ("T.E.") of about 2.8. The bulk viscosity of the polymer was measured at a stress of about 10^4 dynes/cm^2. Measurements were performed at 100°C using procedures as described in W. Graessley, G. Ver Strate, Rubber Chem & Tech, 53 842 (1980) incorporated herein by reference. A strip of polymer (1 x 10 x 1.2 cm) is clamped at one end and allowed to extend under gravitational stress. The extension rate (dl/dt) is calculated as a function of the density and Newtonian viscosity, and it is assumed that Troutons Rule, $3\eta_{shear} = \eta_{elongation}$ applies. The bulk viscosity of the polymer was found to be a typical value for

1   an ethylene-propylene-5-ethylidene-2-norbornene terpolymer
2   of the same molecular weight, i.e. 4×10^5 poise.Table III.
3   This bulk viscosity is too low to permit satisfactory processing in a
4   commercial elastomer plant. Such polymers exhibit such severe cold
5   flow problems that the polymer rapidly agglomerates as a single solid
6   mass and is not readily removed from the recovery vessels.

TABLE II

Process Variable	Run # A	Run # B	Run # C
Residence Time min	17	15	13
Temperature °C	27	27	27
Pressure Kp a	413	413	413
Total hexane feed kg/h	7.5	24.2	31.6
ethene kg/100 kg hexane	3.4	3.86	2.5
propene kg/100kg hexane	11.0	12.0	6.8
ethylidene norbornene kg/100kg hexane	0.65	0.74	.0156
$VOCl_3$ catalyst m mole/hr	2.07	13.4	15.3
$Al_2Et_3Cl_3$ cocatalyst m mole/hr	12.4	80.6	92.1
transfer agent ppm on ethylene	400	200	125
Cationic Agents			
$EtAlCl_2$ m mole/hr	–	–	46
HCl	–	80.4	–

EXAMPLE II

The polymerization reaction of Example I was repeated in substantially the same manner using the conditions set forth in Run B of Table II. Although the polymer found had substantially the same Mooney Viscosity and thickening efficiency as the polymer of Run A, its bulk low strain rate viscosity was higher than the high molecular weight control (Table III).

EXAMPLE III

The polymerization reaction of Example I was repeated using the conditions of Run C (Table II). Again an oil soluble polymer of substantially lower T.E. is produced with improved SSI as compared to the high molecular weight control (see Sample D of Table III below). Yet the bulk viscosity is nearly as high as the high molecular weight control. In this example $EtAlCl_2$ was used as the cationic initiator whereas HCl was used in Example II. If desired a promoter of this invention can be used with the $EtAlCl_2$. An analysis for unsaturation detected 0.2 weight percent ethylidene norbornene. The polymer is substantially saturated.

The polymers of this invention Run B (Example II) and Run C (Example III) are compared to Run A (Example I) a low molecular weight polymer as a control, and a high molecular weight commercially available ethylene-propylene polymer as an additional control. The results are shown in Table III. The high molecular weight polymer exhibits poor shear stability (SSI = 30%). While the low M.W. control (Run A) has a good SSI value (18%), it has a low bulk viscosity (4×10^5 poise). As a result it can not be readily handled because of severe agglomeration problems. The polymer of Run B is substantially identical to the branched control polymer (Run A) except that its bulk viscosity is 1.3×10^6, and therefore, can be readily handled. It forms a crumb which remains as discrete particles for a time sufficient to empty the recovery vessel and complete polymer finishing and packaging. While the polymer of Run C has a slightly higher SSI (23%) it is still acceptable.

The shear stability index (SSI) is determined by measuring the initial viscosity of the polymer, subjecting it to sonic shear and then again measuring the viscosity. The percent change in viscosity, expressed as a percent value, is the SSI.

The thickening efficiency (T.E.) of the polymers of Runs A, B and C are all within acceptable limits. The polymers were tested for T.E. measurements by dissolving them in a solvent extracted neutral mineral lubricating oil having a viscosity of 150-SUS at 37.8°C.

TABLE III

Comparison of Polymers

Polymer	wt.% ethylene	Mooney 100°C	$[\eta]$*	η_o poise	SSI (%)	T.E.
A low molecular weight control	44	15	1.4	4×10^5	18	1.8
C —	43	20	1.6	1.3×10^6	23	2.2
B —	45	23	1.4	4.0×10^6	18.5	1.9
D high molecular weight control	44	45	2.0	2×10^6	30	2.8

* intrinsic viscosity in decalin at 135°C.

EXAMPLE IV

The experiment of Example II is rerun in the same manner except that isobutene is fed to the reactor at the same rate as 5-ethylidene-2-norbornene. The polymer has a bulk viscosity in excess of 10^7 poise at a strain rate of ca 10^{-3} sec at 100°C. There is less than 1.5×10^{-3} moles unsaturation/100g polymer.

It is not intended that the scope of this invention be limited by the method of manufacture. While the Examples refer to a continuous flow stirred reactor, any method of polymerization suitable for ethylene copolymer polymerization may be used. For example, a tubular reactor of the type utilized in the manufacture of polyethylene may be used.

In carrying out the polymerization of this invention in a tubular reactor, all of the catalyst system need not be introduced simultaneously. The Ziegler catalyst can be introduced at the reactor inlet and the cationic initiator can be introduced downstream after polymerization has commenced.

As used in the specification and claims, the term "substantially saturated" means that the polymer has less than 5.0×10^{-3} moles of olefinic unsaturation/100 g. polymer. Preferably the unsaturation level is less than 10^{-3} moles/100 g of polymer.

The polymer prepared by the method of this invention are oil soluble polymers which are useful as viscosity modifiers. They may be used with any class of lubricating fluids in which they are soluble, either alone, or in conjunction with other oil additives. The term "lubricating fluid" as used in the specification and claims means naphthenic, aromatic or paraffinic petroleum oil fractions which are generally suitable for use as lubricating fluids as well as synthetic lubricating oils such as polyesters, poly-alphaolefins of C_5-C_{20} alphaolefins and C_{10} trimers. The polymer of this invention are preferably utilized in the lubricating fluid at about 0.5% to about 5% by weight of the overall composition, more preferably from 0.8 to 1.5% by weight.

The polymers of this invention have a bulk viscosity which is at least 3 times that of a linear ethylene-propylene polymer of the same intrinsic viscosity and ethylene content.

CLAIMS:

1.   A lubricating oil composition comprising a lubricating fluid and an effective amount of a thickening agent which comprises a substantially saturated, long chain, branched ethylene ter- or tetrapolymer comprising ethylene, an alpha-olefin, a non-conjugated diene monomer having a first site of unsaturation which is coordination catalyst polymerizable and a second site of unsaturation which is cationically polymerizable, and optionally, a cationically polymerizable mono-olefin; said polymer having a bulk viscosity at $100^{\circ}C$ and strain rate less than 10^{-3} sec^{-1} which is at least 3 times that of a linear ethylene-propylene polymer having the same intrinsic viscosity and ethylene content.

2.   The composition of Claim 1 wherein the alpha-olefin is a C_3-C_{18} alpha-olefin.

3.   The composition of Claim 2 wherein the alpha-olefin is a C_3-C_8 alpha-olefin.

4.   The composition of Claim 3 wherein the alpha-olefin is propylene.

5.   The composition of claim 1, 2, 3 or 4 wherein

the non-conjugated diene is 2-methyl hexadiene-1,5; 2-methyl heptadiene-1,6; 5-methylene-2-norbornene, 5-ethylidene-2-norbornene, 5(2'-methyl-1-propene)-2-norbornene, 5-methyl vinyl-2-norbornene, 3-methallyl cyclopentane; or 3(2'-methyl-1-propene) cyclopentene or dicyclopentadiene.

6.   The composition according to Claim 5 wherein the non-conjugated diene is 5-ethylidene-2-norbornene.

7.   The composition according to Claim 1 wherein the alpha-olefin is propylene and the non-conjugated diene is 5-ethylidene-2-norbornene.

8.   The composition according to any one of the preceding claims wherein the cationically polymerizable monomer is isobutene.

9.   The composition according to Claim 1 wherein the alpha-olefin is propylene   the non-conjugated diene is 5-ethylidene-2-norbornene , and isobutene is present as the cationically polymerizable mono-olefin.

10.   The composition according to Claim 9 wherein the polymer has isobutene and 5-ethylidene-2-norbornene   incorporated in equal molar quantities.

11.   The lubricating oil composition according to any one of the preceding claims wherein the polymer is present at from 0.5% to 3% by weight of the composition.

12.   The lubricating oil composition according to Claim 11 wherein the polymer is present at from 0.8% to 2.0% by weight.

13.   A process for preparing a polymer of ethylene, an alpha-olefin, and a non-conjugated diene which has a first site of unsaturation which is coordination catalyst polymerizable and a second site of unsaturation which is cationically polymerizable which comprises utilizing a catalyst system to initiate polymerization wherein the catalyst system comprises:

        (a) a coordination catalyst; and

        (b) a cationic polymerization catalyst.

14.   The process according to Claim 13 wherein the coordination catalyst comprises a Ziegler catalyst.

15.   The process according to Claim 14 wherein the Ziegler catalyst comprises a metal compound selected from the group consisting of VCl_4, $VOCl_3$, $TiCl_4$ and $Ti(OR)_4$ wherein R is an alkyl group of one to eight carbon atoms, and a cocatalyst selected from the group consisting of (1) a trialkyl aluminum compound where the alkyl is a C_1-C_8 alkyl or (2) a compound having the general formula $R'_mAl_nX_p$ wherein X is halogen, R' is an alkyl group of one to eight carbon atoms, n is an integer, $m+p=3n$ and $m \geq p$.

16.   The process according to Claim 15 wherein X is chlorine or bromine.

17.   The process according to Claim 15 or 16 wherein R' is ethyl.

18.   The process according to Claim 15 wherein the cocatalyst is Et_2AlCl or $Et_3Al_2Cl_3$.

19. The process according to any one of claims 13 to 18 wherein the cationic polymerization catalyst is (1) a cationic initiator which in conjunction with the cocatalyst is a cationic polymerization initiator, (2) a cationic promoter which in conjunction with the cocatalyst is a cationic polymerization initiator or (3) a cationic initiator which in conjunction with a cationic promoter is a cationic polymerization initiator.

20.   The process according to Claim 19 wherein the cocatalyst is $Et_3Al_2Cl_3$ and the cationic polymerization initiator is $EtAlCl_2$.

21.   The process according to Claim 19 wherein the cocatalyst is Et_2AlCl and the cationic promoter is a tertiary alkyl halide or a benzyl halide.

22.   The process according to Claim 19 wherein

23. . The process according to Claim 22 wherein the cationic promoter is t-butyl chloride or benzyl chloride.

24. The process according to Claim 19 wherein the cocatalyst is $AlEt_3$ or Et_2AlCl or $Al_2Et_3Cl_3$ and the cationic initiator is HCl.

25. The process according to any one of claims 15 to 24 wherein the metal compound is $VOCl_3$ or VCl_4.

26. The use of a polymer prepared by the process according to any one of claims 13 to 25 as a viscosity modifier for a lubricating fluid.